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(54) ELECTRICAL DEVICE AND METHOD OF MAKING SAME

(71) We, OWENS-ILLINOIS INC., a corporation organized and existing under the laws of the State of Ohio, United States of America, of Toledo, State of Ohio, United States of America, (Assignee of RAYMOND LOUIS DIETZ and JAMES JOSEPH TILLMAN), do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention relates to electrical devices and to their manufacture and more particularly relates to improved die attach pastes.

The art is aware of electrical devices, for example DIPs in which a die, i.e. a silicon chip which carries an integrated circuit on an upper portion thereof, is adhesively bonded to an electrically insulative support by means of epoxy resins, the latter means being commonly referred to as a die-attach. The epoxy resins which are usually applied by, for example, screen printing techniques may include effective filling amounts of inorganic fillers.

The present invention is directed to improved electrical devices of the type described above wherein the die attach means comprises the product obtained by heat curing a solvent-soluble further-curable organopolysiloxane consisting essentially of the hydrolysis and partial condensation product obtained by a process comprising: Heating a silane of the formula $CH_3Si(OR)_3$, or a mixture of silanes of the formula $CH_3Si(OR)_3$, and $C_6H_5Si(OR)_3$ in a mole ratio of the latter to the former of between 1:10 and 10:1, wherein R is an alkyl of 1-6 carbon atoms, with water in an amount of 1.5 to 10 moles of water per mole of total silane, and in the presence of an effective catalytic amount of an acid hydrolysis catalyst, from 1 to 10 hours between ambient up to, and including, the reflux temperature to form a solution of a hydrolyzed, siloxane partial condensation product; and concentrating the solution by heating to remove some but not all volatile material including alkanol by product and water so as to obtain a solution of a hydrolyzed further partially condensed, solvent-soluble organopolysiloxane. Suitably the concentrating will be done to produce a solids content of about 62% to about 75% by weight.

Preferably, the solvent-soluble further-curable organopolysiloxane is a solidified material which is produced by precuring the concentrated solution of the hydrolyzed, further partially condensed, solvent-soluble organopolysiloxane by heating below the gel point thereof and then solidifying, as by flaking, the resultant liquid solvent-soluble further-curable organopolysiloxane. These organopolysiloxanes are available commercially and may be manufactured in accordance with the teachings of U.S. Patent Nos. 3,389,121, 3,389,114 and 3,414,540 all of which are hereby incorporated by reference. A particularly suitable organopolysiloxane is the solvent-soluble further-curable organopolysiloxane produced from a mixture of phenyltriethoxysilane and methyltriethoxysilane wherein the molar amount of the former is greater than the molar amount of the latter, with an especially suitable molar ratio being about 4:1. Quite outstanding materials will be obtained by heating, to effect hydrolysis and partial condensation, a mixture of about 2-3 moles of water per mole of total silane, and about 1-10 parts per million of HCL based on the combined weight of water and silane.

In a preferred embodiment of this invention the means for bonding the silicon chip to the support will also include the product obtained by heat curing the heat-curable solvent-soluble organopolysiloxane in admixture with effective filling amounts of particulate inorganic fillers. Preferably the fillers will be of a size less than about 35 microns. These fillers may take the form of being at least one

metal preferably, copper, iron, or nickel which will be employed in an effective thermal conductivity enhancing amounts, i.e. they will be employed in amounts sufficient to enhance the removal of heat when the electrical device is operated and/or the inorganic fillers may include effective thermal stabilizing amounts and effective thermal expansion adjusting amounts of such inorganic fillers as refractory oxides, minerals, clays and diatomaceous earth. Exemplary of such suitable fillers, which not only may serve to adjust the thermal expansion of the bonding means to be compatible with the silicon chip and support but which also may be employed to adjust the consistency, or viscosity, of the material, as applied, to be compatible to the particular method of application, are such fillers as mica, aluminium silicate, titania, aluminum oxide, silicon dioxide, zirconium oxide, zircon, magnesium oxide, spinel, calcium, magnesium or zinc carbonates, or silicates, and the like. Preferably the weight ratio of the inorganic fillers to the organopolysiloxane will be less than about 9:1 and usually, when employed, the fillers will be present in a weight ratio to the organopolysiloxane of at least about 3:7.

While generally there will be no need to do so, if desired, the material which is applied for bonding the silicon chip to the support may include effective cure promoting amounts of a cure promoter. Preferably such promoters include phenylphosphonic acids, amine compounds, for example, gamma aminopropyltriethoxysilane, and methylated and isobutylated melamine formaldehyde partial condensate resins, as generally set forth in U.S. Patent No. 3,935,346 which is hereby incorporated by reference. Usually the amount of the cure promoter will be about 15% by weight, or less, based on the organopolysiloxane. If desired primers may be employed on either the silicon chip or the support but generally this will not be necessary. Exemplary of suitable primers include the thermoset acrylics and aminosilanes as generally set forth in U.S. Patent Nos. 3,707,397 and 3,650,808.

Some of the advantages of the present invention are that the composition which is employed to bond the silicon chip to the electrically insulative support has a long pot life, it is a one part system, unlike the epoxy system, it is curable in short periods of time, no out-gassing results upon utilization and is capable of withstanding temperatures up to around 400°C, it is non-reactive with the surroundings, is non-flammable, and can be conveniently, expeditiously and economically employed.

The organopolysiloxanes contemplated herein have been used as coatings on such materials as plastics, metals and glass. In this respect reference may be had, for example, to U.S. Patents 3,451,838, 3,457,221 and 3,460,980. Additionally they have been employed to produce laminates, and, in this respect reference may be had to U.S. Patent No. 3,654,058. None of these patents, however, are directed to forming electrical devices as contemplated herein.

The composition which is applied to the electrically resistive support is applied in a substantially anhydrous carrier, which is an organic solvent for the solvent-soluble further-curable organopolysiloxane. A wide variety of solvents will be routinely selected but particularly suitable solvents are those sold under the tradename "Carbitol", or under the tradename "Cellosolve", or mixtures thereof. Generally the solvents are conventional organic polar solvents, e.g. alkanols, ketones, ethers and esters. Particularly suitable are diethyleneglycol monobutyl-ether acetate and ethyleneglycol monobutyl-ether or mixtures thereof. When employed the fillers will be simply admixed with the solvent solution of the organopolysiloxane. The composition may be applied by conventional techniques, with screen printing being especially highly preferred. When employing screen printing in addition to the organopolysiloxane and the solvent therefor the composition will preferably include an amount of inorganic fillers which is sufficient to adjust the viscosity, or consistency, of the material so as to make it conducive to being applied by screening. Usually a viscosity of about 200,000 cps. will be quite suitable for this purpose. Obviously if effective cure promoting amounts of a cure promoter are employed they may be added in any suitable manner into the composition.

After being applied to the support the composition is usually slightly heated to evaporate the solvent but the heating is insufficient to cure the further-curable organopolysiloxane; then the die, or silicon chip, having the integrated circuit is applied to the residual material and then subsequently the assembly is heated for a time and at a temperature sufficient to cure the organopolysiloxane to a thermoset condition. Suitable results will be obtained by generally employing a heating cycle in which the temperature is between about 160°C. to about 250°C. for a period of time of between about 15 or 30 seconds to about 10 or 15 minutes. After the material has been screen printed onto the electrically insulative support, i.e. into the recess 11, quite conveniently the procedure thereafter will be employed using a die attach apparatus applied commercially by Unitec Die Bonder as their Model 8140-04-12. While the above sets forth the present invention to enable those skilled in the art to make and use same there nonetheless follows further exemplification.

The invention will now be further described by reference to the drawing which schematically illustrates an embodiment of this invention.

Referring to the drawing the embodiment

illustrates a portion of an electrical package commonly referred to in the art as a DIP. This package comprises an electrically insulative, or resistive, support generally designated 10, typically formed of an inorganic material such as a ceramic, for example alumina or beryllia, which is provided with a central recess 11. Disposed at the lower surface of recess 11 is the product 12 obtained by heat curing the solvent-soluble further-curable organopolysiloxane as contemplated herein. This organopolysiloxane serves to adhesively bond the under surface of the silicon chip, generally designated 13, having an integrated circuit thereon to support 10 in recess 11. Simplifiedly and schematically, the integrated circuit is represented by the general raised grid portions 13a which represents the circuitry interconnecting diffused circuitry in the silicon chip, e.g. N and/or P regions, formed in the silicon chip.

EXAMPLE I

A solvent-soluble further-curable organopolysiloxane which is commercially available as a flaked solid from Owens-Illinois Inc., under their designation Type 908 was employed. This material is a solidified solvent-soluble further-curable organopolysiloxane manufactured from a mixture of phenyltriethoxysilane and methyltriethoxysilane in a molar ratio of about 4:1 (former to the latter). The silanes are hydrolyzed and condensed by employing about 2-3 parts per million, based on the total weight of silane and water, of HCl and between about 2-3 moles of water per mole of total silane. Generally the material is produced in accordance with the teachings of U.S. Patent No. 3,414,540. The solvent-soluble further-curable organopolysiloxane was then dissolved in diethyleneglycol monobutylether acetate to a solids concentration of about 70% by weight. To this solution copper powder (generally having a particle size of about 20 microns) was added in a weight ratio of about 8:1 to about 8.5:1 based on organopolysiloxane solids. The material was intimately mixed to produce a paste and this paste was then screened into the recess of an alumina support conventionally used in the production of DIP electrical devices. The paste was applied using a 165 mesh screen and was then dried at about 100°C. for 7-10 minutes. The alumina support with the residual bonding material was then placed on a heater block which was preheated to about 160°C. and was allowed to remain there for about 5-10 seconds and then the silicon chip was rubbed into the paste over a period of about 2-3 seconds. The unit with the silicon chip having the integrated circuit, which chip is in intimate contact with the further-curable organopolysiloxane composition, was then heated at a temperature of about 160°C. for about 10 minutes whereby the organopolysiloxane became thermoset and provided excellent bonding of the silicon chip into the recess of the electrically insulative ceramic.

While the foregoing describes the present invention it will be, of course, apparent that modification is possible which pursuant to the patent laws and statutes do not depart from the spirit and scope thereof.

WHAT WE CLAIM IS:

1. An electrical device comprising an electrically insulative support, a silicon chip carrying an integrated circuit and means for bonding said chip to said support, said means comprising the product obtained by heat curing a solvent-soluble, further-curable organopolysiloxane consisting essentially of the hydrolysis and partial condensation product obtained by a process comprising: heating a silane of the formula $CH_3Si(OR)_3$, or a mixture of silanes of the formula $CH_3Si(OR)_3$ and $C_6H_5Si(OR)_3$ in a mole ratio of the latter to the former of between 1:10 and 10:1, wherein R is an alkyl of 1-6 carbon atoms, with water in an amount of 1.5 to 10 moles per mole of total silane, and in the presence of an effective catalytic amount of an acid hydrolysis catalyst, from 1 to 10 hours between ambient up to, and including, the reflux temperature to form a solution of a hydrolyzed siloxane partial condensation product and concentrating the solution by heating to remove some but not all volatile material including alkanol by product and water so as to obtain a solution of a hydrolyzed, further partially condensed solvent-soluble organopolysiloxane.

2. A device as claimed in claim 1, in which R is ethyl.

3. A device as claimed in claim 1 or 2 in which said means further includes the product obtained by heat curing said organopolysiloxane in admixture with effective filling amounts of inorganic fillers.

4. A device as claimed in claim 3 in which said filler comprises effective thermal conductivity enhancing amounts of at least one thermally conductive metal.

5. A device as claimed in claim 3 or 4, in which the weight ratio of filler to said organopolysiloxane is less than about 9:1.

6. A device as claimed in claim 3, 4 or 5, in which said filler comprises effective thermal stabilizing amounts and effective thermal expansion adjusting amounts of inorganic fillers.

7. A device as claimed in any one of the preceding claims, in which said means is the product obtained by heat curing said organopolysiloxane in the presence of effective cure enhancing amounts of a cure promoter.

8. A device as claimed in claim 1, substantially as hereinbefore described with reference to, and as illustrated in the accompanying drawing.

9. A device as claimed in claim 1, substantially as hereinbefore described in the foregoing Example.

10. A method for manufacturing an electrical device which comprises applying a material to an electrically insulative support, said material being capable upon application of heat of bonding a silicon chip carrying an integrated circuit to said support, said material comprising a solvent-soluble further-curable organopolysiloxane consisting essentially of the hydrolysis and partial condensation product obtained by a process comprising: heating a silane of the formula $CH_3Si(OR)_3$ or a mixture of silanes of the formula $CH_3Si(OR)_3$ and $C_6H_5Si(OR)_3$, in a mole ratio of the latter to the former of between 1:10 and 10:1, wherein R is an alkyl of 1-6 carbon atoms, with water in an amount of 1.5 to 10 moles of water per mole of total silane and in the presence of an effective catalytic amount of an acid hydrolysis catalyst from 1 to 10 hours between ambient up to, and including, the reflux temperature to form a solution of a hydrolyzed siloxane partial condensation product and concentrating the solution by heating to remove some but not all volatile material including alkanol by product and water so as to obtain a solution of a hydrolyzed, further partially condensed solvent-soluble organopolysiloxane.

11. A method as claimed in claim 10, in which said material further includes in admixture with said solvent-soluble further-curable organopolysiloxane effective filling amounts of inorganic fillers.

12. A method as claimed in claim 10, in which said material further includes effective cure enhancing amounts of a cure promoter.

13. A method as claimed in claim 10, substantially as hereinbefore described with reference to, and as illustrated in the accompanying drawing.

14. A method as claimed in claim 10, substantially as hereinbefore described in the foregoing Example.

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1 SHEET *This drawing is a reproduction of the Original on a reduced scale*